



Consumer and  
Corporate Affairs Canada

Consommation  
et Corporations Canada

(11) (A) No. 1 232 854

(45) ISSUED 880216

(52) CLASS 196-1  
C.R. CL. 196-17

(51) INT. CL. C10G 1/04<sup>4</sup>

(19) (CA) **CANADIAN PATENT** (12)

(54) Use Of a Submersible Viscometer in The Primary  
Separation Step Of the Hot Water Process For  
Recovery Of Bitumen From Tar Sand

(72) Schramm, Laurier L.,  
Canada

(73) Granted to Alberta Energy Company Ltd.  
Canada

Canadian Occidental Petroleum Ltd.

Canada

Esso Resources Canada Limited

Canada

Gulf Canada Limited

Canada

Majesty (Her) the Queen, in right of the Province of  
Alberta, as represented by the Minister of Energy  
and Natural Resources

Canada

HBOG-Oil Sands Limited Partnership

Canada

PanCanadian Petroleum Limited

Canada

Petro-Canada Inc.

Canada

(21) APPLICATION No. 480,350

(22) FILED 850429

NO. OF CLAIMS 2

**Canada**

DISTRIBUTED BY THE PATENT OFFICE, OTTAWA  
CCA-274 (11-82)

480380

1  
2  
3  
4

"USE OF A SUBMERSIBLE VISCOMETER IN  
THE PRIMARY SEPARATION STEP OF  
THE HOT WATER PROCESS FOR  
RECOVERY OF BITUMEN FROM TAR SAND"

5

ABSTRACT OF THE DISCLOSURE

6

The hot water process is controlled in response to

7

viscosity measurements taken in situ in the middlings in the primary

8

separation vessel. The viscosity in the middlings is found to vary.

9

Therefore, the layer of maximum viscosity is located and the viscosity

10

at this depth is monitored. Adjustments are made to the process to keep

11

this maximum viscosity below a pre-determined limit.

1                                    FIELD OF THE INVENTION

2                    This invention relates to an improvement of the flotation-  
3 sedimentation step, for recovering bitumen from a tar sand slurry in  
4 a primary separation vessel, which step forms part of a conventional  
5 tar sand plant circuit. More particularly, it relates to the manner  
6 in which the viscosity of the middlings is measured and to the  
7 utilization of the measurements so obtained to guide adjustments to  
8 the process conditions.

9                                    BACKGROUND OF THE INVENTION

10                   Tar sands, also referred to as oil sands and bituminous  
11 sands, contain a heavy oil usually referred to as bitumen.

12                   There are tar sand deposits, in the Athabasca region of  
13 Alberta, which are today being commercially exploited. In connection  
14 with these operations, the tar sand is first mined and the bitumen is  
15 then extracted from the mined tar sand by a process called the hot water  
16 process. The extracted bitumen is subsequently upgraded by refinery-  
17 type processing, to produce synthetic crude.

18                   The tar sand is a mixture of sand grains, connate water,  
19 fine minerals of the particle size of clay, and bitumen. It is commonly  
20 believed that the connate water envelopes the grains of sand, the fine  
21 solids are distributed in the water sheaths, and the bitumen is trapped  
22 in the interstitial spaces between the water-sheathed grains.

23                   The hot water process is now well described in the patent  
24 and technical literature. A schematic of the circuit is shown in  
25 Figure 3.



1 In broad summary, this process comprises first conditioning  
2 the tar sand, to make it amenable to flotation-sedimentation separation  
3 of the bitumen from the solids. Conditioning involves feeding mined tar  
4 sand, hot water (180°F), an alkaline process aid (usually NaOH), and  
5 steam into a rotating horizontal drum, wherein the ingredients are agitated  
6 together. Typically, the amounts of reagents added are in the following  
7 proportions:

8 tar sand	- 3250 tons
9 hot water	- 610 tons
10 NaOH	- 4 tons (20% NaOH)

11 Enough steam is added to ensure an exit temperature of the mixture  
12 from the drum of about 180°F. The residence time in the drum is  
13 typically about 4 minutes.

14 During conditioning, the mined tar sand (in which the  
15 bitumen, connate water and solids are tightly bound together) is  
16 converted into an aqueous slurry of porridge-like consistency, wherein  
17 the components are in loose association.

18 The slurry leaving the drum is screened, to remove oversize  
19 material, and then flooded or diluted with additional hot water. The  
20 diluted slurry typically comprises 7% by weight bitumen, 43% water, and  
21 50% solids. Its temperature is typically 160 - 180°F.

22 The diluted slurry then is transferred to the primary  
23 separation step, wherein it is temporarily retained in a large separation  
24 vessel having a cylindrical upper section and conical lower section. (This  
25 vessel is hereafter referred to as the "PSV" - for 'primary separation  
26 vessel'.) The vessel is similar to a thickener and has a rake system  
27 in its lower end, to assist in discharging the sand bed which accumulates  
28 there. The slurry is retained in the PSV for about 45 minutes in a  
29 quiescent condition.

1                   During this interval, air bubbles, incorporated into the  
2 dilute slurry during conditioning, attach themselves to the bitumen, which  
3 is in the form of flecks or globules. Most of the aerated globules are  
4 buoyant and they rise through the slurry, to collect at the upper surface  
5 in the form of a froth. This froth is referred to as primary froth.

6                   Most of the coarse solids, primarily being sand particles,  
7 sink through the slurry, are concentrated in the conical bottom end of  
8 the vessel, and are discharged through a bottom outlet. This stream  
9 is discarded as tailings (known as the 'primary tailings').

10                  Not all of the bitumen becomes sufficiently aerated so  
11 as to rise and join the primary froth. Some of this non-buoyant  
12 bitumen is lost with the primary tailings. Most of it, together with a  
13 large part of the fines, collects in the mid-section of the PSV. This  
14 aqueous mixture is termed "middlings".

15                  A dragstream of the middlings is withdrawn from the  
16 vessel and is fed into subaerated flotation cells, wherein secondary  
17 separation is practised. Here the middlings are subjected to  
18 vigorous agitation and aeration. Bitumen froth, termed "secondary  
19 froth", is produced.

20                  Typically, the primary and secondary froths have the  
21 following compositions:

	<u>Primary (% by weight)</u>	<u>Secondary (% by weight)</u>
22                  Bitumen	66.4	23.8
23                  Solids	8.9	17.5
24                  Water	24.7	58.7

1           It will be noted that the secondary froth is considerably  
2 more contaminated with water and solids than the primary froth. One  
3 seeks to minimize this contamination, as the froth stream requires  
4 downstream treatment, to remove solids and water, before it can be fed  
5 to the upgrading process.

6           It is therefore desirable to operate the process so that  
7 as much of the bitumen as possible reports to the primary froth.

8           In summary then, the contents of the PSV may be described  
9 as existing in the form of three sequential layers. At the base, one  
10 has the tailings - this is primarily sand with some water and a minor  
11 amount of bitumen entrained therein. Above this layer, one has the  
12 middlings - this is water containing fines and insufficiently buoyant  
13 bitumen. But passing downwardly through the middlings are many coarse  
14 sand particles and rising through the layer are some buoyant bitumen  
15 globules. And at the top, one has the froth.

16           Of particular interest are the well-aerated bitumen  
17 globules, which should rise and form the primary froth, which is the  
18 main commercial product of the process. These globules must make  
19 their way up through the middlings.

20           If the middlings are too viscous, the well-aerated bitumen  
21 globules may fail to achieve the needed upward velocity, and may end  
22 up being discharged with the primary tailings or being withdrawn  
23 with middlings for treatment in the secondary separation circuit. If  
24 the globules exit with the primary tailings, they are lost entirely  
25 from the process. If they are removed to secondary recovery, they  
26 will be recovered in the form of poor quality froth.

1232854

1                   At this point, it is appropriate to point out: (1) that  
2 the nature of the tar sand feed is variable; and (2) that the capability  
3 of the hot water process to extract the contained bitumen is significantly  
4 affected by the nature of the tar sand feed.

5                   More particularly, the tar sand may contain a relatively  
6 high content of bitumen and a relatively low content of fines. This type  
7 of feed is referred to as "rich" tar sand. Alternatively, the tar sand  
8 may be relatively low in bitumen and high in fines. Such a feed is  
9 referred to as "lean" tar sand.

10                   Typically, a "rich" tar sand can have a composition as  
11 follows:

12	14.44%	bitumen
13	0.36%	water
14	85.2%	total solids

15                   Typically, a "lean" tar sand can have a composition as  
16 follows:

17	7.56%	bitumen
18	0.5%	water
19	91.84%	total solids.

20                   The percentage fine solids ( $-44\mu$  solids in the total  
21 solids) can range from 5% for rich tar sands to as high as 25% for some  
22 lean tar sands.

23                   In general, the rich tar sand feeds yield high primary  
24 froth recoveries. The lean feeds give low primary froth recoveries.  
25 Stated otherwise, the lean feeds are difficult to process with the hot  
26 water extraction procedure; they do not contain much bitumen and such  
27 bitumen as they do contain is difficult to extract.

1                   This is partly because the lean feeds contain many fines,  
2   which interfere with the flotation-sedimentation separation taking place  
3   in the middlings layer of the PSV. In addition, the flecks or globules  
4   of bitumen which appear in the PSV middlings, when lean tar sand is  
5   the feed, are minute compared to the globules that are there when the  
6   tar sand feed is rich. These minute flecks do not rise as readily as  
7   the larger flecks.

8                   If the fines content in the middlings becomes high, the  
9   flotation mechanism can literally become inoperative. There is so little  
10   primary froth being produced that the process performance is unacceptable.  
11   In this instance, the contents of the PSV may have to be jettisoned and  
12   the process started up again.

13                  There are a number of courses of action open to the  
14   operator by which he may adjust and alleviate undesirable process conditions  
15   in the PSV arising from the nature of the tar sand feed. For example, he  
16   can:

- 17                   - adjust the rate of NaOH addition; or
- 18                   - adjust the rate of water addition to the conditioning
- 19                    or flooding steps; or
- 20                   - blend some better quality tar sand in with the lean
- 21                    tar sand, to provide a blended feed; or
- 22                   - vary the residence time or temperature in the
- 23                    conditioning drum.

24                  A crucial matter, though, is to know when to make these  
25   adjustments and to what extent the adjustment should be made. This  
26   requires that a process parameter be monitored, which parameter gives  
27   the operator a useful guide on which to base the adjustments.



1           It has heretofore been broadly taught in the prior art  
2   that the viscosity of the middlings can be monitored and maintained  
3   within staged ranges, to optimize the primary bitumen froth recovery  
4   from the PSV. This teaching appears in Canadian patent 889,823, filed  
5   by Graybill et al. Also of interest are Canadian patents, 889,825  
6   and 841,581.

7           However, in accordance with conventional practise, the  
8   viscosity has been monitored in one of the following ways:

- 9           - withdrawing a sample from the middlings dragstream  
10           and measuring the sample viscosity with an appropriate  
11           instrument; or
- 12           - lowering a sampler into the middlings, taking a grab  
13           sample, and measuring the sample viscosity with an  
14           appropriate instrument; or
- 15           - applying density measurements to either of the  
16           foregoing samples and assuming that the viscosity  
17           varies proportionately with the density.

18           Now, there are certain shortcomings associated with  
19   these prior art practises.

20           If one samples the middlings dragstream, one must assume  
21   that this sample - taken at one level of the PSV (there is usually  
22   only a single outlet in the PSV wall) - is representative of the entire  
23   column of PSV middlings.

24           When one attempts to measure the viscosity of this sample,  
25   one is dealing with a mixture of sand, oil, clay, and water. The sand  
26   and oil begin to settle and rise instantaneously. In addition, the  
27   mixture is not static. It is impossible to duplicate the flow and  
28   turbulence conditions which exist within the PSV.

1 Perhaps for these reasons, the industry has moved toward  
2 measuring the density of the sample and assuming that the trend of  
3 viscosity will follow the trend of density.

4 SUMMARY OF THE INVENTION

5 In the fundamental step of this invention, the viscosity  
6 of the middlings is taken in situ in the PSV with a submersible viscometer.

7 In the testing which led up to this invention, when this  
8 was done the following discoveries were made:

- 9 (1) that the viscosity varies strikingly at various  
10 depths in the middlings in the PSV;
- 11 (2) that while the in situ-measured viscosity in the  
12 PSV may vary significantly, the density of the  
13 middlings when measured in connection with grab  
14 samples may vary very little - therefore there does  
15 not appear to be a useful correlation between the  
16 two that may be relied on; and
- 17 (3) that the viscosity measurements obtained in situ  
18 vary significantly from those obtained by taking  
19 grab samples at the same depth in the PSV and  
20 measuring the viscosity of the grab samples in a  
21 conventional instrument external of the PSV.

1           Stated otherwise, it has been found that it is necessary to  
2   measure the viscosity of the middlings in the dynamic environment of the  
3   PSV contents, in order to obtain reliable and useful measurements. It is  
4   postulated that the currents which arise in the PSV (from the continuous  
5   entry of fresh slurry, the withdrawals of the tailings and middlings streams,  
6   and the influences of dropping solids and rising bitumen), together with the  
7   presence of the solids at the point of testing, combine to create a unique  
8   and depth-variable viscosity regime in situ which differs in kind from that  
9   which may be measured in grab samples and dragstreams. It is this unique  
10   in situ viscosity regime which must be monitored in order to give the desired  
11   guidance for process control.

12           In a preferred embodiment, one may "hunt" out the maximum  
13   viscosity level in the middlings in the PSV by moving the submersible  
14   viscometer vertically and taking measurements at different levels. One  
15   then alleviates the undesirable process conditions by monitoring  
16   viscosity at this level and making one or more process adjustments,  
17   as previously described, to control said maximum viscosity and bring it  
18   close to a pre-determined desired value.

19           Broadly stated, the invention is an improvement in the primary  
20   separation step of the hot water process for extracting bitumen from tar sand  
21   in a primary separation vessel, wherein the bitumen floats upwardly in a  
22   tar sand slurry to form a froth layer, the coarse solids drop to form a  
23   tailings layer, and a middlings layer is formed between the froth and the  
24   tailings. The improvement comprises: providing a submerged viscometer in  
25   the middlings layer and actuating said viscometer to measure the viscosity  
26   of the middlings at one or more levels in the vertical column of middlings  
27   and produce signals, external of the vessel, which are indicative of said  
28   measurements; taking sufficient measurements to determine the viscosity of  
29   the region of maximum viscosity within the middlings layer; and adjusting  
30   the viscosity of the middlings in response to said signals to maintain the  
31   maximum viscosity in the column below a predetermined value, whereby the

**1232854**

- 1 flotation of the bitumen through the middlings layer to the froth layer is
- 2 substantially enhanced.

DESCRIPTION OF THE DRAWINGS

Figure 1 is a partially sectional side view of the viscometer used in connection with the invention;

Figure 2 is a sectional side view showing the viscometer suspended in the PSV of the pilot hot water process circuit used in developing the invention;

Figure 3 is a schematic showing the hot water process circuit;

Figure 4 is a plot of measured in-situ viscosity versus depth in the PSV at which the viscosity was measured, showing the variation in viscosity which is present in the PSV middlings at different levels, for a single tar sand feed treated in two ways - one without NaOH addition and the other with NaOH;

Figure 5 is a plot of measured density values for grab samples taken at different depths for the tar sand runs which generated the data for Figure 4; and

Figure 6a is a fanciful representation of the PSV contents during the run in which NaOH was not used;

Figure 6b is a fanciful representation of the PSV contents during the run in which NaOH was used.

- 1
- 2
- 3
- 4
- 5
- 6
- 7
- 8
- 9
- 10
- 11
- 12
- 13
- 14
- 15
- 16
- 17
- 18
- 19
- 20
- 21
- 22
- 23
- 24
- 25
- 26
- 27
- 28
- 29
- 30
- 31

11  
12  
13  
14  
15  
16  
17  
18  
19  
20  
21  
22  
23  
24

15  
16  
17  
18  
19  
20  
21  
22  
23  
24

25  
26  
27  
28  
29  
30  
31

30

31

C

1           The PSV 7 was part of a circuit illustrated in Figure 3.  
2   This circuit comprised a tumbler 9, in which tar sand was mixed with  
3   hot water, NaOH, and steam, and conditioned. The product slurry from  
4   the tumbler 9 was diluted with additional hot water in a pump box 10.  
5   The diluted slurry from the pump box 10 was transferred into the PSV 7  
6   and retained there under quiescent conditions, to produce bitumen, froth,  
7   tailings, and middlings. Middlings were withdrawn from the PSV 7 and  
8   treated in a bank of sub-aerated flotation cells 11, to produce secondary  
9   froth and secondary tailings. The foregoing steps were conducted in  
10   accordance with conventional hot water process conditions.

11   Example 1

12           The pilot circuit was used to process a tar sand  
13   designated "A". This was known to be a poorly processing, lean feed.  
14   Two runs were made during which the feed was treated by the hot water  
15   process. One run was carried out with NaOH process aid having been  
16   incorporated in the slurry; the other run was carried out without NaOH.  
17   Viscosity measurements were made during each run using the viscometer 1  
18   at different depths in the middlings in the PSV 7. Two curves or plots  
19   of measured in situ viscosity versus depth were developed. Plot 1 in  
20   Figure 4 involved the run without NaOH. Plot 2 in Figure 4 involved  
21   the run with NaOH. The details of the conditions and primary froth  
22   recovery results of the two runs are now set forth.

23           Tar Sand "A" composition:

24           9.8% bitumen

25           3.2% water

26           87.0% solids

27           21.3% fine solids (expressed as % of -44 $\mu$  solids  
28           in the total solids)

Pilot Processing of Oil Sands "A"

Oil Sand Feed Rate - 630 g/s  
 Slurry Temperature - 80°C  
 Rate of Total Water Addition - 418 g/s

<u>NaOH Addition (wt. %)</u>	<u>Primary Bitumen Recovery (%)</u>
0.000	9.5
0.025	22.1

As shown by plot 1 for the run without NaOH, at a depth of about 0.4 m in the PSV, the viscosity measured with the viscometer was about 15 mPa.s. As the viscometer was lowered, the viscosity increased rapidly to 110 mPa.s. at a depth of 0.8 m, and then diminished to about 80 mPa.s. at a final depth of about 1.2 m.

Thus the PSV contents, when the PSV was operating on this lean tar sand A, were shown to be characterized by:

- a low viscosity at the upper end of the body of contents (as very little primary bitumen froth was generated by the poorly processing slurry in the absence of NaOH);
- changes in viscosity with depth;
- and a "plug" or layer of high viscosity middlings intermediate its ends.

The PSV contents were visually observed through the glass wall of the vessel. Figure 6a depicts what was observed. Again, there was only a thin layer of primary bitumen froth at the top end of the vessel contents and a viscous intermediate layer, which contained much bitumen.



1           The same tar sand A was then treated under the same  
2 conditions as the Plot I run, except that in this second run a  
3 conventional amount of NaOH was used. The in-situ viscosity versus  
4 depth results are shown by Plot II in Figure 4. At the top of the  
5 cell contents the viscometer 1 indicated a high viscosity (130 mPa.s.),  
6 indicative of the thick bitumen froth layer which was produced. As the  
7 viscometer was lowered to 0.3 m, it passed through the froth-middlings  
8 interface and the measured viscosity dropped off sharply. The viscometer  
9 1 indicated that the viscosity continued to decline to a limiting value  
10 around 10 mPa.s. in the lower part of the vessel. There was no "plug"  
11 of highly viscous middlings to hinder the rise of the bitumen globules.  
12 An improved primary bitumen froth recovery was obtained in this run  
13 as compared with the first run. Visual inspection during the run  
14 indicated that the PSV contents were of the form shown in Figure 6b.  
15 There was a thick froth layer and no noticeable viscous layer laden  
16 with bitumen.

17           Thus there was correlation between the results indicated  
18 by the in situ viscometer measurements and PSV performance as indicated  
19 by the primary oil recoveries.

20           During the two runs, several grab samples were also taken  
21 at depths corresponding with some of those at which the viscometer 1  
22 took in situ measurements. Attempts to measure viscosity representative  
23 of conditions within the PSV, on withdrawn samples, resulted in failure.  
24 The above-noted problems, that is, the ascent of bitumen in the sample  
25 jars, the rapid settling of coarse solids, and the impractical require-  
26 ments for reproducing the flow and turbulence currents of the PSV,  
27 caused such measurements to be abandoned.

1 In summary, these results show that:

- 2 (1) use of the submersible viscometer produces results  
3 that indicate that there are viscosity changes that  
4 occur within a PSV with depth;  
5 (2) If high viscosity layers are developed in the PSV  
6 middlings, they do trap bitumen and diminish primary  
7 bitumen froth production; and  
8 (3) These high viscosity layers can be eliminated by  
9 adjusting process conditions, thereby improving  
10 primary bitumen froth recovery.

11 In use, the signals emitted by the viscometer 1, submerged  
12 in the middlings, are monitored and the viscosity of the middlings  
13 are adjusted by altering one of the aforesaid process conditions, to  
14 maintain the maximum viscosity in the middlings column below a pre-  
15 determined value.

1 THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY  
2 OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

3 1. In the primary separation step of the hot water process for  
4 extracting bitumen from tar sand in a primary separation vessel, wherein the  
5 bitumen floats upwardly in a tar sand slurry to form a froth layer, the coarse  
6 solids drop to form a tailings layer, and a middlings layer is formed between  
7 the froth and the tailings, the improvement comprising:

8 providing a submerged viscometer in the middlings layer and  
9 actuating said viscometer to measure the viscosity of the middlings at one or  
10 more levels in the vertical column of middlings and produce signals, external  
11 of the vessel, which are indicative of said measurements;

12 taking sufficient measurements to determine the viscosity of the  
13 region of maximum viscosity within the middlings layer;

14 and adjusting the viscosity of the middlings in response to said  
15 signals to maintain the maximum viscosity in the column below a predetermined  
16 value,

17 whereby the flotation of the bitumen through the middlings layer  
18 to the froth layer is substantially enhanced.

19 2. The improvement as set forth in claim 1 comprising:

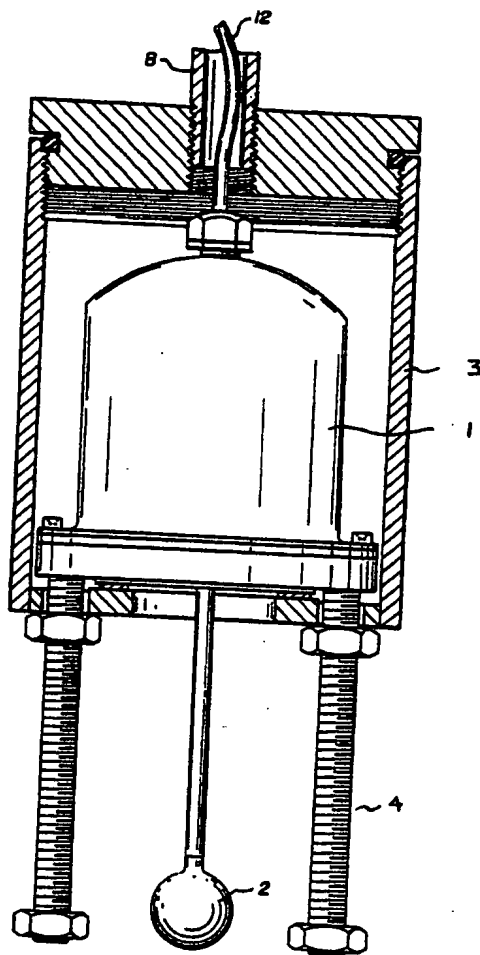
20 moving the viscometer vertically within the column of middlings  
21 and locating and measuring the viscosity of the layer of middlings which  
22 has the maximum viscosity.



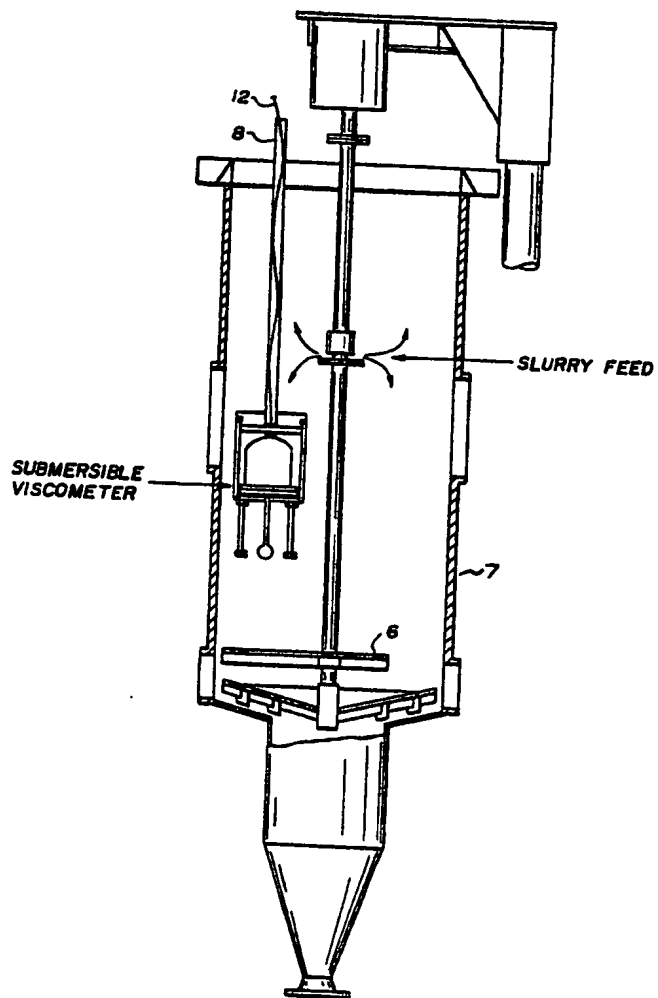
1232854

6-1

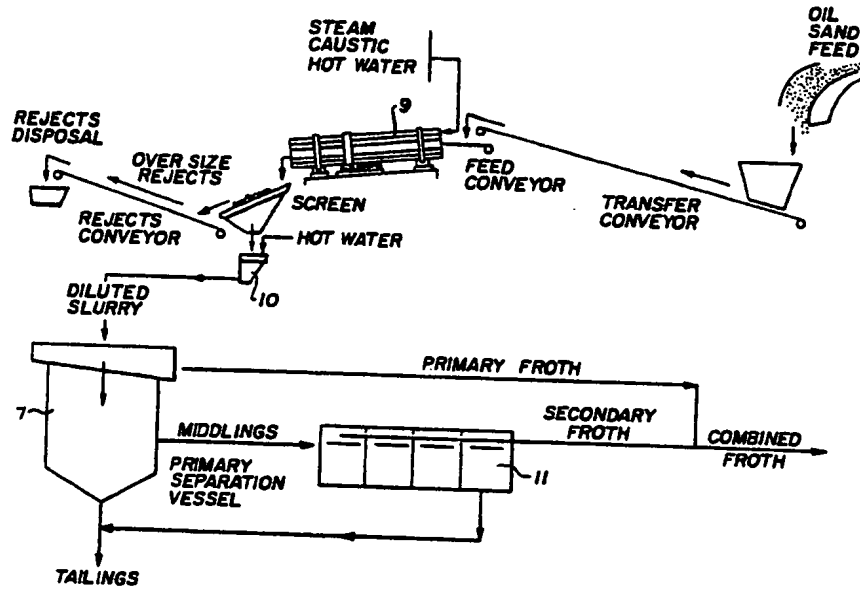
Fig. 1.



Patent agent:  
EP Johnson

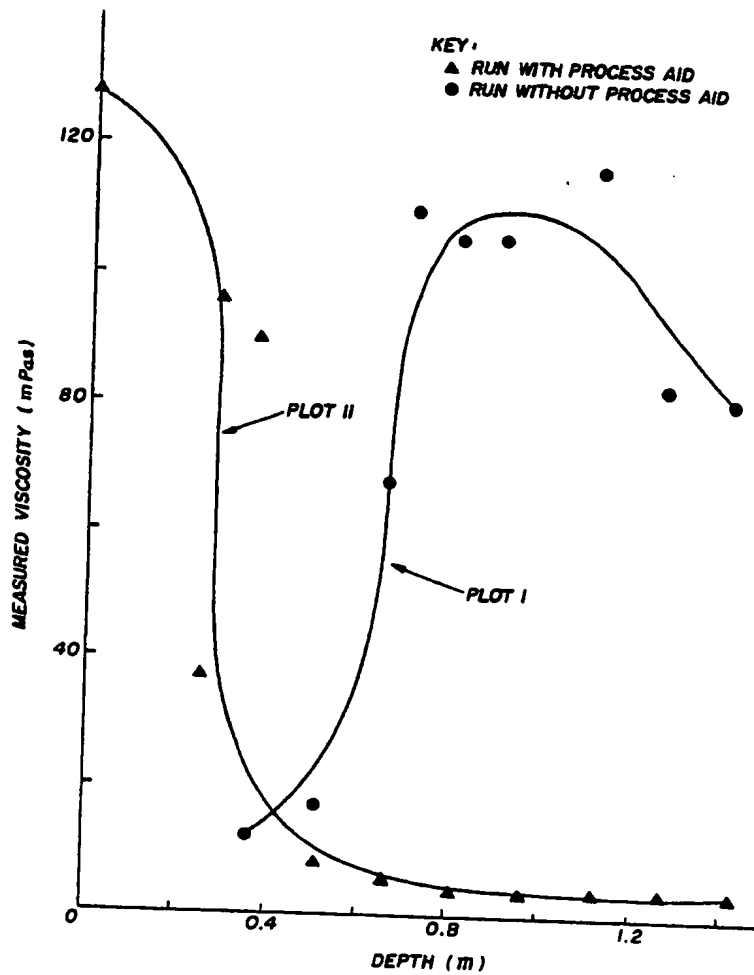
Fig. 2.

Patent agent:  
EP Johnson

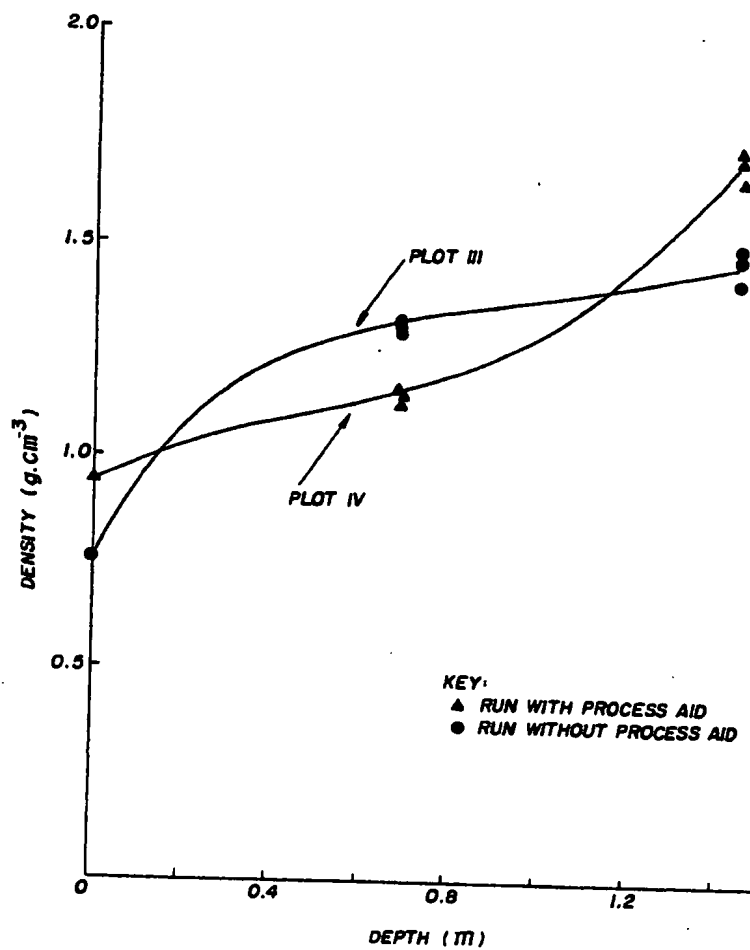
Fig. 3.

Patent agent:  
E. P. Johnson

6-4

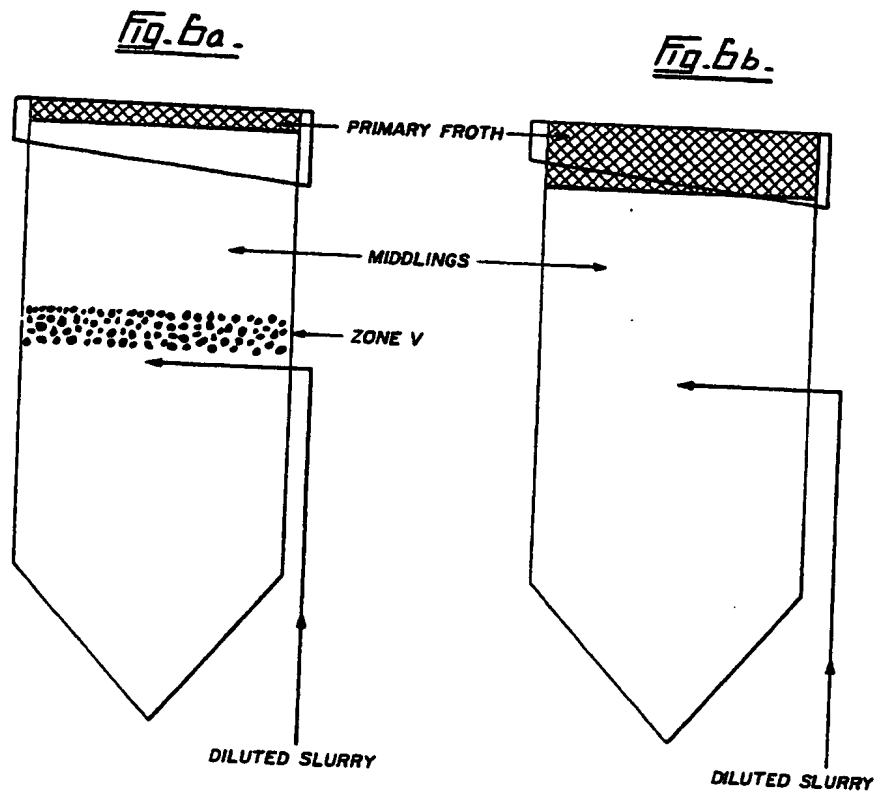
Fig. 4.

Patent agent:  
EP Johnson

Fig. 5.

Patent agent:  
EP Johnson





Patent agent:  
EP Johnson